

# Hydrophobic Particle Films: A New Paradigm for Suppression of Arthropod Pests and Plant Diseases

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**ABSTRACT** We introduce a unique concept, hydrophobic particle film technology, that represents the combined synthesis of knowledge on the use of hydrophobic films, physical particle barriers, and white reflective surfaces to suppress arthropod pests and diseases of agricultural crops. The hydrophobic particle film is based on the inert mineral, kaolin, that is surface treated with a water-repelling agent. We demonstrate suppression of important tree fruit arthropod pests and diseases by altering the plants surface with dust applications of these hydrophobic particles. There are a number of potential mechanisms involved in pest suppression. Arthropods can be repelled from, or infestations suppressed, on a plant coated with a hydrophobic particle film barrier by making the plant visually or tactually unrecognizable as a host. In addition, insect movement, feeding, and other physical activities can be severely impaired by the attachment of particles to the arthropods body as they crawl upon the film. Diseases can be prevented by enveloping the plant in a hydrophobic particle film barrier that prevents disease inoculum or water from directly contacting the leaf surface. Thus, many fungal and bacterial pathogens that require a liquid film of water for disease propagule germination are prevented. Other key features of the hydrophobic particle film are that it reduces heat stress by reflecting sunlight with its bright white color, and does not affect plant photosynthesis or productivity because of the porous nature of the film. Our results suggest that the hydrophobic particle film concept could offer broad spectrum protection against arthropod pests and diseases in certain agricultural crops.

**KEY WORDS** inert pesticide, mineral particle barrier, kaolin

CHEMICAL PESTICIDES THAT act as neurological or physiological toxins are used extensively in agricultural crop production to control damage by arthropod pests and diseases. Public concerns for human health and the environment have led to an effort to reduce pesticide use by using an integrated pest management (IPM) approach to pest control. Currently, the U.S. Environmental Protection Agency is scrutinizing the registration of a number of chemical pesticides based on toxicity and residue concerns. The inevitable loss of some key chemical pesticides, compounded by microbial and insect resistance to pesticides, emphasizes the need for developing alternatives to toxic chemical pesticides.

From the 1920s to 1960s, considerable research was conducted on mineral particles to identify those that had insecticidal properties and to determine the mechanisms of insecticidal action. The major body of this research was conducted on stored grain and structural pests because they had the greatest potential of

being controlled by dust applications. Extensive screening of a wide array of minerals by Alexander et al. (1944) on granary weevil larvae, by David and Gardiner (1950) on 4 species of larvae and adult grain and flour beetles, and Ebeling and Wagner (1959) on the termite *Kaolitermes minor* (Hagen) concluded that very hard nonsorptive particles (e.g., diamond, quartz) or soft porous sorptive particles (silica oxide, aluminum oxide) were the most effective killing agents. Effectiveness generally increased as particle size decreased to an ideal size of 1-2  $\mu\text{m}$  because of improved adherence to the insect cuticle. The primary mechanisms of action were the partial removal of the insect's outer cuticle (epicuticle) through abrasion by hard nonsorptive particles (Kalmus 1944, Wigglesworth 1944, David and Gardiner 1950) or structural disruption of the epicuticle by adsorption of epicuticular lipids to sorptive particles (Alexander et al. 1944, Hunt 1947, Ebeling and Wagner 1959). Both mechanisms induced rapid water loss from the insect's body and caused death by dessication. Consequently, there was an inverse relationship between insect mortality and relative humidity, and mineral particles were virtually ineffectual at 100% RH. Plugging of the insect hindgut by inert particles after being ingested was another possible mechanism causing death of the spotted cucumber beetle, *Diabrotica undecimpunctata howardi* Barber (Richardson and Glover 1932), and

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walnut husk fly, *Rhagoletis completa* Cresson (Boyce 1932).

Since the 1960s, there has been little interest in inert minerals as insecticides because of the availability of cheap and effective synthetic materials. Other reasons why agricultural research on dusts has been neglected are that they have been implicated in reducing plant productivity and inducing arthropod pest outbreaks on plants. Trees and plants along dirt roads that become covered by road-dust have been known to be susceptible to phytophagous mite and scale outbreaks because the dust inhibits their natural enemies (Debach 1979). Mineral particles originating from quarrying, open-pit mining and road traffic are also known to decrease plant productivity and increase aphid pests and fungal infections (Farmer 1993). Foliar applications of kaolin have been shown to be phytotoxic by damaging cuticle of the leaf and increasing water loss (Eveling 1972, Eveling and Eisa 1976). More recent research on mineral particles as pesticides has been limited to formulations containing particles impregnated with synthetic pesticidal compounds (Kirkpatrick and Gillenwater 1981, Margulies et al. 1992) or microbial agents (Studdert et al. 1990, Tapp and Stotzky 1995).

Mineral based whitewashes have been examined for the prevention of insect vectored transmission of viral plant diseases. White reflective surfaces repel certain aphids by affecting their host-finding and settling responses (Kennedy et al. 1961, Kring 1962). Whitewashes come in various forms and are generally composed of kaolinite, bentonite, and attapulgite with the addition of spreading and sticking agents that are designed to whitewash the plant foliage or soil surrounding the plant (Nawrocka et al. 1975; Bar-Joseph and Frenkel 1983; Marco 1986, 1993). This approach has had success that is limited to repelling aphid and leafhopper vectors.

Research on the use of inert mineral particles as control agents for foliar bacterial and fungal diseases has also been lacking. Most research on foliar diseases has focused on the use of mineral compounds, such as sodium carbonate, which is caustic and modifies the pH of the plant surfaces under moist conditions. Thus, the germination and growth of fungal diseases is prevented (Olivier et al. 1998). One of the most commonly used mineral materials for foliar fungal diseases in agriculture today is bordeaux, a mixture of caustic and pH modifying hydrated lime and the toxic heavy metal, copper sulfate.

Kaolin is a white, nonporous, nonswelling, nonabrasive fine grained platy aluminosilicate mineral ( $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ ) that easily disperses in water and is chemically inert over a wide pH range. Coating grade kaolin is >90% pure and has a high brightness quality of >85%. Mined kaolin has traces of 2 metals,  $\text{Fe}_2\text{O}_3$  and  $\text{TiO}_2$ ; the former must be removed to obtain white brightness qualities >85% that is required for various industrial applications (Harben 1995). Technical advances within the past decade have made it possible to produce kaolin particles with specific sizes, shapes and light reflective properties. Kaolin particles are engi-

neered with specific properties for use in the paper, paint, cosmetic, and plastic industries have been largely ignored by the agricultural industry. Certain uses require that the particles be made with varying degrees of hydrophobicity by coating them with waterproofing agents such as chrome complexes, stearic acid and organic zirconate using new technologies. These developments have opened new possibilities for the use of mineral particles for pest control in agriculture.

There has been virtually no research on the foliar applications of hydrophobic mineral particles for disease and insect suppression in agricultural crops. We hypothesize that insect pests and diseases of plants can be prevented by coating plants with a hydrophobic mineral particle film. This particle film coating would serve as a physical barrier that would conceivably repel arthropods, or suppress infestations, by making the plant visually or tactually unrecognizable as a host. Further, we propose that insect movement, feeding, and other physical activities can be severely impaired by the attachment of particles to the arthropods body. Many fungal and bacterial plant pathogens require a liquid film of water for propagule germination and direct contact with the leaf surface. Although the cuticular waxes of leaves are somewhat hydrophobic, water in the form of dew or rain is still able to wet the leaf (Holloway 1970). In theory, plant infecting pathogens of this type could be prevented by coating plants with a hydrophobic particle film that would serve as a physical barrier to liquid water. Therefore, diseases would be prevented by disrupting the linkage of water with the leaf surface. Fungal and bacterial diseases could also be prevented by the physical particle barrier that keeps the pathogen propagule from contacting the leaf surface. Because of the porous nature of this particle film, we propose that both insects and diseases can be controlled with hydrophobic particle films without interfering with transpiration or photosynthesis. Laboratory, greenhouse, and field research focused on the hydrophobic kaolin particle, M-96-018, to determine if our new concept was viable for insect and disease suppression in agricultural crops.

### Materials and Methods

The hydrophobic particle used in these studies was M96 (M-96-018 Kaolin), which is composed of kaolin processed to a bright white color of >85%, sized to  $\leq 2 \mu\text{m}$ , and made hydrophobic by coating it with a proprietary synthetic hydrocarbon (Engelhard, Iselin, NJ).

In laboratory and greenhouse experiments, thin uniform coatings of M96 were applied to plant foliage and other plant parts by a camel's-hair brush 4–10 mm wide. Known amounts of M96 ( $0.2 \mu\text{g M96}/\text{cm}^2$  leaf surface) were lightly brushed onto the leaf surfaces until a uniform smooth white film was produced that made the foliage visibly white. This method was useful for experiments where only a few leaves of a plant needed to be coated.

Field applications of M96 were made by using a Solo backpack sprayer model 423 with a duster kit (Solo, Newport News, VA) that was calibrated to deliver a known amount (grams) per tree. Enough material was used to entirely coat the foliage of the tree with a white uniform dusting of M96. Direct measurements of particle densities ( $\mu\text{g}/\text{cm}^2$ ) were made by washing the leaves with methanol into aluminum weighing pans, evaporating off the methanol, and weighing the particles that remained. Particle applications applied by camel's-hair brush or backpack duster produced hydrophobic films that ranged from 85 to 200  $\mu\text{g}$  M96/ $\text{cm}^2$  leaf surface. These amounts varied because of differential particle adhesion to the leaves of the various plant species examined (e.g., smooth pear leaves versus relatively rough and pubescent apple leaves).

The particle film was quantified ( $\mu\text{g}/\text{cm}^2$  leaf area) in some experiments by an indirect calibration that used the L-value from a Minolta ChromaMeter (model CR221, Minolta, Ramsey, NJ). The L-value is a measurement of whiteness that ranges from black to white. A calibration curve was derived by coating green colored paper discs (1 cm diameter) that had a range of different weights of M96 particles. The initial L-value of a disc was measured before particle treatment, and a known amount of particles was applied and the final L-value was again measured. Particle amount ( $\mu\text{g}/\text{cm}^2$ ) was then regressed against the difference between the initial and final L-value (termed delta L-value). The prediction equation was  $y$  ( $\mu\text{g}/\text{cm}^2$ ) = (delta L-value) $^2$  ( $R^2 = 0.97$ ,  $n = 48$ ,  $P = 0.01$ ). This calibration was validated by selecting 48 leaves from apple, *Malus domestica* (L.) Borkh, with varying amounts of M96 particles. The delta L-value was measured and the weight of M96 per leaf was determined ( $\mu\text{g}/\text{cm}^2$ ) by washing the leaves with methanol into aluminum weighing pans, evaporating off the methanol, and weighing the residues. In the field, the L-values of treated leaves were measured and then the particle film was removed by washing, after which the L-value was remeasured. The delta L-value was converted into  $\mu\text{g}/\text{cm}^2$  particles per leaf area using the prediction equation.

**Arthropod Studies.** In a laboratory experiment, the influence of a hydrophobic particle film on pear psylla, *Cacopsylla pyricola* Foerster, host and ovipositional preference was examined. Twenty adults were confined in an arena by a polycarbonate cylindrical cage (14.8 cm diameter by 60 cm tall) and given a choice between untreated and M96 treated potted pear seedlings, *Pyrus communis* L., that had all but the last 2 fully expanded terminal leaves removed. M96 was applied to the top and bottom surfaces of the leaf by camel's-hair brush method. The experiment was a completely randomized design with 9 replications that was conducted in a growth chamber maintained at 23°C and a photoperiod of 15:9 (L:D) h. Adult and egg numbers that were present on the leaf surfaces of each seedling were recorded 5 d after adult introduction into the arena. Data on adult and egg numbers were analyzed by analysis of variance (ANOVA) and treatment means were compared using the least significant dif-

ferences (LSD) method,  $\alpha = 0.05$  (SAS Institute 1995).

In a no-choice experiment, survival and oviposition for pear psylla adults were determined by confining adults on pear leaves treated with M96, untreated leaves, and on a glass slide that represented no food source. Application of M96 and the environmental conditions were the same as that in the pear psylla choice experiment. Eight adults  $\approx$  2 wk of age were confined to treated or untreated leaves by a clip-cage (Puterka and Peters 1988) that was modified by increasing the chamber size from the original 6 mm i.d. to 19 mm i.d. Only the 1st or 2nd fully expanded leaves of terminal growth were used on potted pear seedlings. The experiment was a completely randomized design with 9 replications. Adult survival and egg numbers were recorded 1 and 3 d after introduction to the treatments. Data on adults and oviposition were analyzed by ANOVA and treatment means were compared using the LSD method,  $\alpha = 0.05$  (SAS Institute 1995).

The effect of a hydrophobic particle film on the spirea aphid, *Aphis spireacola* Patch, on apple was also examined in a no-choice experiment. A continuous film of M96 was applied to leaves of apple seedlings ( $\approx$  10 leaves per plant) by camel's-hair brush in the same manner as for pear, and then infested with 10 adult aphids per plant. Aphid numbers were compared with those on untreated control plants 1, 2, and 3 d after infestation. The experiment was a completely randomized design with 6 replications and was conducted in a growth chamber maintained at 25°C and a photoperiod of 15:9 (L:D) h. Treatment means were compared using a  $t$ -test,  $\alpha = 0.05$  (SAS Institute 1995).

In a no-choice experiment, the effect of hydrophobic particle film treated foliage on the twospotted spider mite, *Tetranychus urticae* Koch, populations on apple was examined. Potted plant seedling size and M96 applications were the same as in the pear studies. Treated and untreated seedlings were each infested with 10 adult mites per plant. Mite numbers were compared with those on untreated control plants 3 wk after infestation. The experiment was a completely randomized design with 6 replications and was conducted in a growth chamber maintained at 25°C, and a photoperiod of 15:9 (L:D) h. Treatment means were compared using a  $t$ -test,  $\alpha = 0.05$  (SAS Institute 1995).

In 1994, a field experiment was conducted to determine the influence of hydrophobic particle films on leafhopper damage mainly caused by the potato leafhopper, *Empoasca fabae* (Harris). Potted MacIntosh apple trees were placed randomly beneath the canopy of 4-yr-old Golden Delicious apple trees in an unsprayed orchard. Twelve potted trees 0.5–0.75 m in height were coated on a weekly basis with  $\approx$  10 grams per tree using a Solo backpack duster. Using this application method, a uniform white film visibly the same as when applied by camel's-hair brush was deposited on the top and bottom of the leaves. Leafhopper damage in the form of curled leaves was compared with untreated controls. The experimental design was completely randomized with 12 replications. Treat-

ment means were compared using a *t*-test,  $\alpha = 0.05$  (SAS Institute 1995).

In 1995, M96 was applied to 3-yr-old 'Seckel' pear trees in June to determine if psylla populations could be suppressed in the field. Trees were dusted with 100 g of M96 using a Solo backpack duster just before adult ovipositional activity and reapplied on 4-d intervals twice. Pilot tests determined that 100 g of M96 completely coated the foliage of a tree 3 m tall by 2 m diameter. Treatments were arranged in a completely randomized design with 6 replications. The number of eggs, nymphs, and adults per 8-inch limb terminal were recorded from 4 limbs per tree at 1 and 10 d after the 1st M96 application. Data on counts were analyzed by ANOVA and treatment means were compared using the LSD method,  $\alpha = 0.05$  (SAS Institute 1995).

**Plant Disease Studies.** A field study was conducted on mature pear, *Pyrus communis* L. 'Bartlett', in 1994 to examine the effect of a hydrophobic particle film on fireblight. The study was designed as paired-limb treatment experiment that had M96 treatments and untreated control replicated 6 times. Limbs were randomly selected and treated with 10 g of M96 applied by a Solo backpack duster when >50% of the blossoms were open. After particle applications, both flowers on the treated and untreated limbs received an aerosol spray of  $10^8$  cfu/ml of *Erwinia amylovora* (Burrill), the causal organism of fireblight. Ten to 36 flowers on each branch received these treatment combinations.

In 1995, the same treatments were applied to branches of 'Smoother' apple in full bloom under the same experimental design and methodology except that the M96 application was made at full bloom, then treated with *E. amylovora*. In both years, blossom infection was measured by observing definite flower necrosis. Numbers of infected blossoms were compared between treatments using a *t*-test,  $\alpha = 0.05$  (SAS Institute 1995).

Prevention of apple scab *Venturia inaequalis* (Cooke) Winter by a hydrophobic particle film was determined in 1994 by placing 0.5–0.75 m potted MacIntosh apple trees beneath the canopy of 4-yr-old Golden Delicious apple trees in an unsprayed orchard that had a history of high levels of apple scab. Twelve potted trees were treated with M96 in the same manner and weekly intervals as in the leafhopper experiment. Natural infections by the apple scab fungus were evaluated 12 wk after treatment by counting the number of leaves with scab lesions per seedling. There were 12 replications per treatment in a completely randomized design. Differences in number of scab-infected leaves between the M96 and untreated control were compared using a *t*-test,  $\alpha = 0.05$  (SAS Institute 1995).

In 1995, the efficacy of a hydrophobic particle film against apple scab was compared with a conventional fungicide program on a block of 8-yr-old 'Bisbee Red Spur Delicious' apple trees (3 m tall by 2.2 m diameter). Treatments included: M96 applied at  $\approx 100$  grams per tree by a Solo backpack duster on a weekly basis; a conventional pesticide program that used both insecticides, miticides, and fungicides using a standard

spray guide recommendations (Anonymous 1995); and an untreated control. The conventional pesticide program included the insecticides Pounce 3.2 EC (emulsifiable concentrate, 3.5 ml [AI]/ha), Guthion 50 WP (wetttable powder, 282.5 g [AI]/ha), Lannate LV (295.7 ml [AI]/ha), Carzol 92 SP (732 ml [AI]/ha) and Omite 30 W (wetttable, 489 g [AI]/ha), and the fungicides Penncozeb DF (dry flowable, 3.4 kg [AI]/ha), Rubigan EC (581 g [AI]/ha), Benlate 50 W (42.5 g [AI]/ha), Ferbam 76 WDG (water dispersible granules, 3.4 kg [AI]/ha), Ziram 76 WP (3.4 kg [AI]/ha), and Captan 50 WP (2.26 kg [AI]/ha). Pesticide applications were made based on insect and disease pest pressures that were determined by a biweekly scouting program. There were 3 trees per treatment with the treatments arranged in a randomized complete block design with 4 replications. Two guard rows separated treatments to safeguard against spray drift. The trees were biennially bearing, in a low yield state, and were highly variable in growth. Consequently, yield and growth are not reported because of the high variation. At the end of the season, 100 leaves from vigorous midcanopy shoots (annual growth) were chosen randomly and the number of leaves with apple scab lesions were recorded. Treatments were compared using ANOVA and treatment means were separated using LSD,  $\alpha = 0.05$  (SAS Institute 1995).

A greenhouse study was conducted in 1995 to determine the mechanism of apple scab prevention by a hydrophobic particle film. The 4 treatments were as follows: (1) upper surface of leaves treated with M96 by camel's-hair brush as explained in previous experiments, and then inoculum placed on leaf [25  $\mu$ l of 146,000 cfu/ml (*V. inaequalis*)]; (2) inoculum (at the level of 1st treatment) was placed on the leaf, allowed to dry for 30 min, and covered with a film of M96 by the camel's-hair brush method that resulted in  $87 \pm 6$   $\mu$ g M96/cm<sup>2</sup> leaf surface; (3) positive control where inoculum (at the level of the 1st treatment) was placed on the leaf but not allowed to dry; and (4) negative control where 25  $\mu$ l sterile water was applied to the leaf. The leaves were held in a folded position with a paper clip to prevent water and inoculum drops from rolling off the leaf. The 4 treatments were applied to potted MacIntosh apple seedlings  $\approx 0.3$  m tall with each treatment randomly assigned to 3 leaves per seedling and replicated 3 times within a growth chamber maintained at 19°C. A misting system kept the relative humidity at 100% to prevent evaporation. The trees remained in the growth chamber for 72 h and were then placed in a greenhouse maintained at  $20 \pm 5^\circ\text{C}$ . Counts of apple scab lesions were made 2 mo later, and data were analyzed using ANOVA with treatment means separated using LSD,  $\alpha = 0.05$  (SAS Institute 1995).

**Horticulture and Related Studies.** The effect of hydrophobic particle films on peach yield was evaluated in 1995 in an 8-yr-old peach orchard 'Loring' and 'Harvester' (trees 2.5 m tall by 2 m diameter). The treatments included a conventional commercial insecticide and fungicide program as a control with application needs determined by pest pressure

(Anonymous 1995) and M96 particle film treatment (100 grams per tree) that began after petal fall and was reapplied weekly 10 times up to harvest. Data were collected from the central 3 trees of each replicate. There were 4 replicates of each treatment in a randomized block design. Canopy air temperatures were measured using an Everest 110 IR thermometer (Everest Intersciences, Testin, CA). Fruit yield was measured by picking fruit over a 2-wk ripening period for each cultivar. Shoot length was measured on 10 vertical shoots randomly chosen from each tree at the end of the growing season. Treatment comparisons for canopy air temperature, yield and shoot growth were made using a *t*-test,  $\alpha = 0.05$  (SAS Institute 1995).

To understand how a hydrophobic particle film altered reflected sunlight, the spectral radiance of solar radiation reflected from the canopy of single Seckel pear trees was compared with untreated pear trees. This experiment was conducted on the same trees used in the pear psylla field experiment that were dusted with M96 in 1995. Spectral radiance was measured in 2-nm increments from 330 to 1,100 nm with a Li-Cor spectroradiometer equipped with a telescopic lens (model LI 1800, Li-Cor, Lincoln, NE). Measurements on the illuminated side of single M96 treated and untreated trees (4 replications) were made near solar noon on a cloudless day with the field of view for the instrument being 1.0 m<sup>2</sup> of tree canopy. Pear trees 3 m tall and 2 m in diameter had canopy densities sufficient to fully intercept the field of view.

Relative leaf photosynthetic rates were measured in apple, peach, and pear trees to determine the effect of increasing densities of hydrophobic particle films. The study was conducted in a growth chamber maintained at 20°C, 70% RH, and a photoperiod of 16:8 (L:D) h, and a light intensity of 900  $\mu\text{mol m}^{-2}\text{s}^{-1}$ . The photosynthetic rate of individual leaves was measured with a Li-Cor 6200 photosynthesis system. Four untreated leaves of each tree species were measured and M96 was applied to the upper and lower leaf surface. The particle density of each leaf was measured with a Minolta chromameter and determined by the L-value method. Increasing amounts of particles were applied and photosynthesis and particle density measured after each application. Relative photosynthesis was calculated as the ratio of initial rate to the rate at each particle level. Particle density was regressed with relative photosynthetic rate and the slope compared with zero.

The relationship of transmitted photosynthetically active radiation to the surface area density of 2 types

Table 1. Mean  $\pm$  SE pear psylla adult and egg numbers 5 d after given a choice between untreated and hydrophobic particle film treated pear seedlings in a growth chamber study

Treatment	Adults/plant	Eggs/plant
Hydrophobic particle film		
Untreated control		

Means within columns followed by the same letter are not significantly different, LSD,  $P > 0.05$  (SAS Institute 1995). Treatments began with 20 adults; adults on the sides of the cages were not counted; means of 9 replications.

of particle films was measured in a growth chamber. A rigid cellophane strip 30 by 30 cm received increasing amounts of sieved limestone dust  $\leq 38 \mu\text{m}$  (all particles passing through a 38- $\mu\text{m}$  sieve) and hydrophobic particles manufactured to  $\leq 2 \mu\text{m}$ . The cellophane platform was placed above a line quantum sensor (Li-Cor model 191SA). Photosynthetically active radiation levels from a fixed light source transmitting light from above the platform were recorded as particles were applied. Four measurements of photosynthetically active radiation were made across the treated platform and averaged. The mass of the particles was calculated by weighing the cellophane platform after each new addition of particles. Surface area density (m<sup>2</sup>/m<sup>2</sup>) was calculated assuming the following: (1) a circular vertical projection of each particle on the platform, (2) a particle diameter of 38  $\mu\text{m}$  for the limestone and 2  $\mu\text{m}$  hydrophobic particles, and, (3) a particle density of 2.63 g/cm<sup>3</sup> for each particle application. Particle surface area density (m<sup>2</sup>/m<sup>2</sup>) was plotted against percentage of photosynthetically active radiation.

## Results and Discussion

**Arthropod Studies.** Plant leaves coated with a hydrophobic particle film had adverse effects on all of the arthropods that were studied. Pear psylla adults did not settle or oviposit on M96 treated leaves when given a choice between M96 treated and untreated foliage in the laboratory (Table 1). Upon termination of this experiment, psylla adults were inspected under a microscope and it was found that all of the adults had tarsal segments covered with kaolin particles. This indicates that the psylla adults attempted to settle on M96 treated leaves, but were then repelled by the treated leaf surfaces. The lack of adult mortalities in this experiment indicated that incidental contact with the hydrophobic particle film was not fatal, although

Table 2. Mean  $\pm$  SE pear psylla adult and egg numbers 1 and 3 d after adults were confined on pear leaves treated with M-96-018 Kaolin hydrophobic particles, an untreated control, and on a glass slide with no food source

Treatment	Day 1		Day 3	
	Adults	Eggs	Adults	Eggs
Hydrophobic particle film		0.0 $\pm$ 0.0b		0.0 $\pm$ 0.0b
Untreated control		25.2 $\pm$ 4.7a		59.3 $\pm$ 16.1a
Glass slide—no food		0.0 $\pm$ 0.0b		—

Means within columns followed by the same letter are not significantly different, LSD,  $P > 0.05$  (SAS Institute 1995). Treatments began with 8 adults; means of 9 replications.



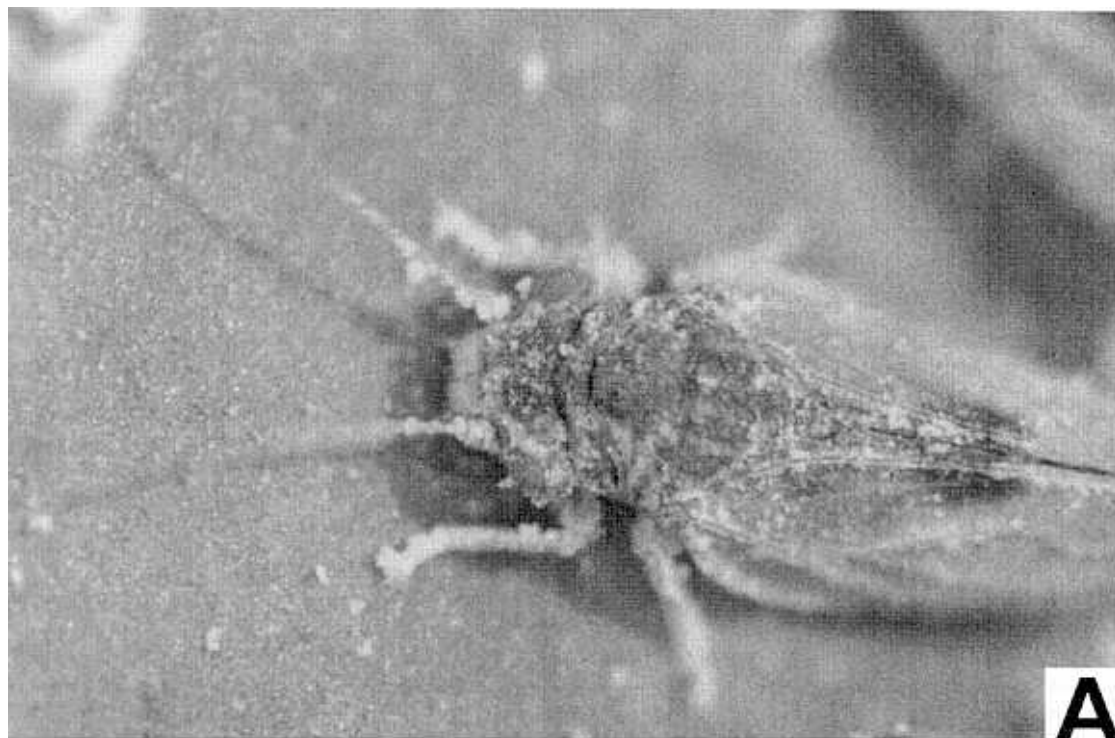


Fig. 1. Pear psylla adult (A) and spirea aphid nymph (B) showing the attachment of hydrophobic particles on their appendages and body 24 h after confinement on a hydrophobic particle film treated leaf.

**Table 3.** Spirea aphid population levels (mean  $\pm$  SE) after adults ( $n = 10$ ) were placed on hydrophobic particle film treated and untreated apple leaves of potted Red Delicious apple seedlings held within a growth chamber

Treatments	No. aphids		
	24 h	48 h	72 h
Hydrophobic particle film	5.3 $\pm$ 1.1b	5.3 $\pm$ 0.9b	5.0 $\pm$ 1.1b
Untreated control	10.8 $\pm$ 1.1a	11.3 $\pm$ 1.0a	13.7 $\pm$ 1.2a

Means within columns followed by the same letter are not significantly different ( $\alpha = 0.05$ ,  $t$ -test). Mean for 6 replications.

the feeding and ovipositional behavior that followed may have been affected.

Pear psylla adults that were confined on hydrophobic particle treated pear leaves in the no-choice experiment did not oviposit and incurred moderate mortalities 24 h after containment on the treated leaf surfaces (Table 2). The adults that remained alive at this time must have been able to feed through the particle coated leaf surfaces, considering the fact that all of the adults contained on a glass slide and deprived of a food source died within 24 h. Over a period of 3 d, nearly all psylla adults died without ovipositing. Other researchers that examined the effects of various inert mineral particles on insects (Alexander et al. 1944, David and Gardiner 1950) considered abrasion and sorption to be key elements for insect killing activity. They found kaolin ineffective as a killing agent because of its relatively soft, nonabrasive and nonsorptive nature compared with the other effective minerals. The psylla adults became heavily coated with hydrophobic particles within 24 h after containment (Fig. 1A). Adults appeared preoccupied by attempts to remove these particles from their body parts or remained motionless compared with untreated adults that actively fed and carried out ovipositional activities. We suggest that the behavior of the adults was disrupted by the clinging particles to the degree that they were unable to feed and eventually starved.

Nearly 50% reduction in spirea aphid numbers occurred within a 24-h period when spirea aphid adults were placed uncaged onto treated apple leaves (Table 3). These populations remained static over a 3-d period on particle film treated apple. Some adult aphids lost footing soon after being placed on particle film treated leaf surfaces, which contributed to initial reductions in number. Lack of nymph production by those aphid adults that became established indicated that either the adults did not reproduce in this un-

**Table 4.** Twospotted spider mite population levels (mean  $\pm$  SE) 3 wk after adults ( $n = 10$ ) were caged onto hydrophobic particle film treated and untreated apple leaves of potted Red Delicious apple seedlings held within a growth chamber

Treatment	No. mites/cage
Hydrophobic particle film	
Untreated control	

Means followed by the same letter are not significantly different ( $\alpha = 0.05$ ,  $t$ -test).

**Table 5.** Mean  $\pm$  SE incidence of *V. inequalis* and leafhopper damage to leaves of potted MacIntosh apple seedlings placed within an apple orchard and treated with dust applications of the M-96-018 kaolin hydrophobic particles, Kearneysville, WV, 1994

Treatment <sup>a</sup>	No. scab lesions/tree	No. leafhopper damaged leaves/tree
Untreated control	32.6 $\pm$ 12.4a	
Hydrophobic particle film	16.5 $\pm$ 7.3b	

Means within columns followed by the same letter are not significantly different ( $\alpha = 0.05$ ,  $t$ -test). Mean for 6 replications.

<sup>a</sup> Treated on a weekly basis 12 times with PF, M-96-018 kaolin, and treatments were evaluated 12 wk after applications began.

suitable environment or the newborn aphid nymphs also became coated with particles (Fig. 1B), lost footing, and fell from the plant.

Twospotted spider mite populations were also reduced by >50% compared with untreated controls (Table 4). Hydrophobic particles were also observed clinging to the mite's bodies and they struggled in an effort to move about the particle treated surface in a similar manner as did psylla adults, which undoubtedly reduced feeding and reproduction. Mite mortalities in this experiment were not noted because of difficulties in discerning mortality. However, mortalities would be expected to occur if mites were not feeding. Additional reductions in mite populations might also be expected by falling from the plant as the aphids did, but this loss could not be determined because the mites were caged on the leaves.

Leafhopper damage in the form of characteristic curled apple leaves was nearly absent in hydrophobic particle film treated apple seedlings placed within an apple orchard (Table 5). Leafhopper numbers were not obtained from this experiment because the original focus of the experiment was apple scab disease suppression. However, leafhoppers, mainly the potato leafhopper, were found to be actively colonizing the untreated trees within the orchard and the same damage resulted, which verified their association with the curled leaf damage on these seedlings. The lack of leafhopper damage on M96 treated apple suggested that they were repelled in the same manner as were pear psylla.

In the field, trees treated with M96 showed immediate reductions in adult psylla numbers 1 d after application (Table 6). The full impact of the hydrophobic particle film treatments on psylla infestations was demonstrated 10 d after application where high reductions in eggs, nymphs, and adults occurred in comparison with untreated controls. These field studies concur with the laboratory studies, and demonstrate that, when given a choice, the primary mechanism of action against arthropod adults is through repellency, which prevents plant infestation. Further, the laboratory studies indicated that reductions >50% could occur in no-choice situations where apterous arthropods that must crawl on the particle film. In doing so, they become entangled by the particle film and fall from the plant or have their feeding activities

**Table 6.** Mean  $\pm$  SE egg, nymph, and adult pear psylla 1 and 10 d after dust applications of M-96-018 kaolin hydrophobic particles in a pear orchard, May 1995, Kearneysville, WV

Treatment	Day 1			Day 10		
	Eggs	Nymphs	Adults	Eggs	Nymphs	Adults
Hydrophobic <sup>a</sup> particle film	158.6 $\pm$ 25.9a	3.0 $\pm$ 2.4a	5.3 $\pm$ 0.9b	52.0 $\pm$ 10.4b	19.5 $\pm$ 3.2b	2.4 $\pm$ 0.4b
Untreated control	171.0 $\pm$ 19.5a	5.7 $\pm$ 1.6a	66.0 $\pm$ 7.3a	284.0 $\pm$ 125.2a	58.0 $\pm$ 15.9a	31.9 $\pm$ 8.1a

Means within a column followed by the same letter are not significantly different (LSD;  $P > 0.05$ ). Mean for 7 replications.

<sup>a</sup>M96-018 kaolin was applied on 25 May and on 31 May 1995 to maintain a visible white coating on the leaves.

severely disrupted to the extent that they could die from starvation. Such situations would occur where mite or aphid colonies may already be present before particle film applications began. In addition, the light reflecting properties of the white hydrophobic particle film coating could alter an arthropods perception of the host plant. Whitewashes have been shown prevent plant viral diseases vectored by aphids (Nawrocka et al. 1975; Marco 1986, 1993) by reflecting sunlight, which alters the aphids host finding and settling behaviors (Kennedy et al. 1961, Kring 1962). Further research is needed to quantify these and other possible mechanisms-of-action that hydrophobic particle films could have on a particular arthropod species. The effects of hydrophobic particle films will likely differ on the myriad of arthropod types, body sizes, and life-stages.

**Plant Pathology Studies.** In a laboratory study, the hydrophobic particle film completely prevented scab inoculum from infecting apple leaves when the inoculum was placed on top of the particle treated leaf and misted with water for 72 h (Table 7). In addition, scab infection was prevented when inoculum was placed on the leaf surface and coated with a hydrophobic particle film, then misted with water for 72 h. This experiment supports our hypothesis that plant infection by pathogens of this type could be prevented by hydrophobic particle films on a leaf surface. These films could either act as a physical barrier to liquid water, thus, disrupt the linkage of water to the leaf surface and prevent infection, or act as a physical barrier to prevent direct contact of the pathogen propagule with water and the leaf surface. The waxy cuticle of most plant leaves lend some hydrophobicity and water contact angles are usually  $>90^\circ$  (Holloway 1970), yet water is still able to adhere to and wet the leaf surface (Fig. 2A). We found that

hydrophobic particle film applications increased leaf hydrophobicity to  $>158^\circ$  (Fig. 2B), which makes water adhesion impossible. Thus, water (e.g., in the form of dew condensation or rain) will be shed from these treated plant surfaces. The photograph of the water droplet in Fig. 2B was only made possible by placing the droplet in a depression on the hydrophobic particle film treated leaf surface.

Antitranspirants, waxes (Kamp 1985, Hagiladi and Ziv 1986, Ziv and Frederiksen 1986, Han 1990, Marco et al. 1994), and silicon films (Bowen et al. 1992) have similar action against foliar diseases, presumably by separating the inoculum from the leaf surface or water, or as a barrier on the leaf surface that prevents hyphal penetration into the plant. Diseases that are controlled by antitranspirants are powdery mildew and leaf rusts (Kamp 1985, Ziv and Frederiksen 1986). Inorganic whitewashes and kaolin applications have also been shown to reduce foliar diseases (Marco et al. 1994). Although the mechanisms for disease suppression by kaolin are not clear, soluble heavy metals contained in the whitewash formulations used by Marco et al. (1994) were suspected. However, natural deposits of kaolin are free of toxic heavy metals, thus, this mechanism is improbable unless these metals were contained in another component of the formulation. Films of potassium silicate provide another mechanism for fungal disease control by forming a crystalline barrier on the leaf surface that physically abrades fungal hyphae (Menzies et al. 1992). Abrasion or heavy metal toxicity is not a likely mechanism for disease suppression by a hydrophobic particle film because it is composed of 99% pure kaolin and is free of heavy metals and is regarded as nonabrasive (1.2 on Moh's scale). Both kaolin and the hydrophobic coating of M96 are chemically inert. Furthermore, the hydrophobic particle film is effective against both bacterial and fungal diseases where physical abrasion of the aforementioned microorganism has not been demonstrated. We verified the nontoxic and nonabrasive nature of the hydrophobic particles by coating *E. amylovora* and *V. inaequalis* colonies cultured on media in petri dishes, which did not affect colony growth (D.M.G. and G.J.P., unpublished data). The mechanism of bacterial and fungal disease control by hydrophobic particle films appears to be most similar to those of antitranspirants, waxes, or silicone films. Yet, hydrophobic particle films offer major advantages over these other materials. The porous nature of the hydrophobic particle film does not impede the gas exchange between

**Table 7.** Effect of M-96-018 kaolin hydrophobic particle film and inoculum position on MacIntosh *V. inaequalis* infection

Treatment	% leaf infection
Inoculum placed on surface of the particle film treated leaf	
Inoculum placed on leaf surface, then coated with a particle film	
Positive control-inoculum on leaf	
Negative control-water placed on leaf	

Means within a column followed by the same letter are not significantly different (LSD,  $P > 0.05$ ).



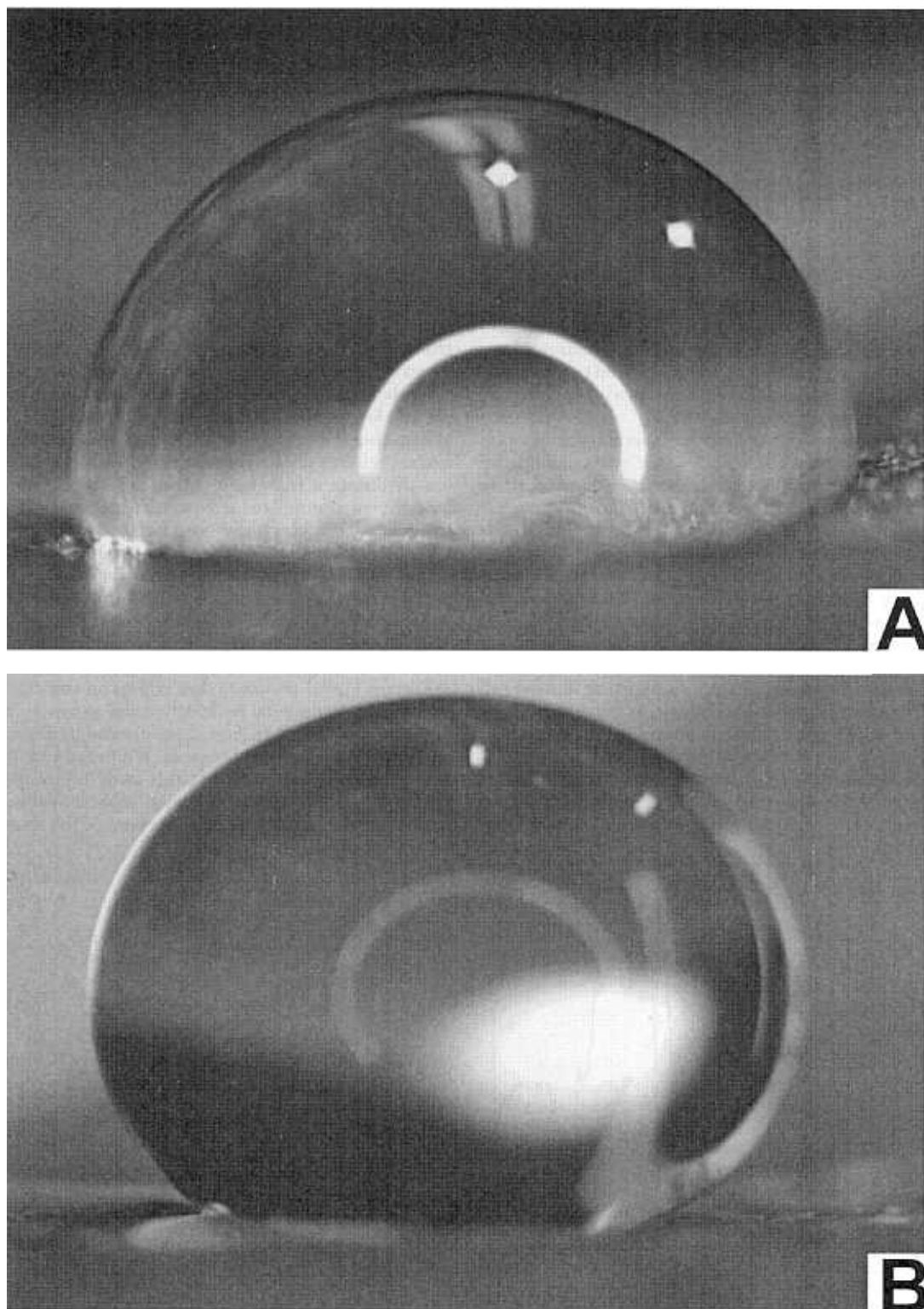


Fig. 2. Water droplet ( $2\ \mu\text{l}$ ) on an untreated (A) and hydrophobic particle film treated (B) peach leaf. The water droplet on the treated leaf (B) is resting in a depression on the leaf to prevent it from rolling off. Contact angle increased from  $95^\circ$  for untreated to  $158^\circ$  for the hydrophobic particle film treated leaf, indicating a substantial increase in water repellency.

Table 8. Effect of M-96-018 kaolin hydrophobic particle film on disease incidence in the field on apple, 1995, Kearneysville, WV

Treatment	% blossom infection by <i>E. amylovora</i>		% leaf infection by <i>V. inaequalis</i>	
	1994 <sup>a</sup>	1995 <sup>b</sup>	1994 <sup>c</sup>	1996 <sup>d</sup>
Hydrophobic particle film			17.0 ± 3.0b	
Untreated control			33.0 ± 9.0a	
Conventional pesticides			—	

Mean within columns followed by the same letter are not significantly different ( $P > 0.05$ , LSD). —, No conventional pesticide treatment for that year.

<sup>a</sup> Bartlett pear. Mean for 6 replications.

<sup>b</sup> Smoothie apple. Mean for 6 replications.

<sup>c</sup> MacIntosh apple. Mean for 12 replications.

<sup>d</sup> Bisbee Red Spur Delicious. Mean for 4 replications.

plants and the atmosphere during transpiration and photosynthesis, which is the primary function of antitranspirants and similar films. This and other effects on the plants physiological processes will be addressed in the next section of this article.

Field studies confirmed the laboratory studies on disease control with hydrophobic particle films by demonstrating ~50% apple scab prevention in particle treated apple seedlings (Table 5) and in apple orchards (Table 8). Other field studies on apple and pear blossoms treated by hydrophobic particle film treatments resulted in significant reductions in fireblight (Table 8). These results suggest that hydrophobic particle films can be effective control agents of plant fungal and bacterial diseases. The lack of complete control in the field was probably attributed to the difficulty in applying a continuous particle film over all of the plant surfaces. Improvements in particle application technology or particle formulation could lead to better control of diseases in the field.

**Horticulture and Related Studies.** Application of the hydrophobic particle film to pear trees in the field

broadly reflects spectral radiance in the visible spectrum (Fig. 3). Yet, measurements of leaf photosynthesis in growth chambers found no reduction in photosynthetic activity in apple, peach, and pear with particle levels up to 3,000  $\mu\text{g}/\text{cm}^2$  leaf surface (Fig. 4). When the slope of particle density was regressed against relative photosynthetic rate it was not significantly different from zero ( $t$ -test,  $\alpha = 0.05$ ). Others have shown a neutral to increased dry matter response to mineral particle applications (Basnizki and Evenari 1975, Stanhill et al. 1976, Moreshet et al. 1979, Lowery et al. 1990). However, Marco (1986) demonstrated a significant reduction in potato yield in applying whitewashes with 15% mineral solids and Hirano et al. (1995) significantly reduced yield and photosynthesis with nonwhite particles on cucumber and bean.

Farmer (1993) reviewed dust effects on vegetation from an environmental perspective and generally reported reduced growth from dusts created by cement and other manufacturing sources. We found that the hydrophobic particles used in this study transmitted photosynthetically active radiation more effectively than limestone dusts, primarily because of the finely,

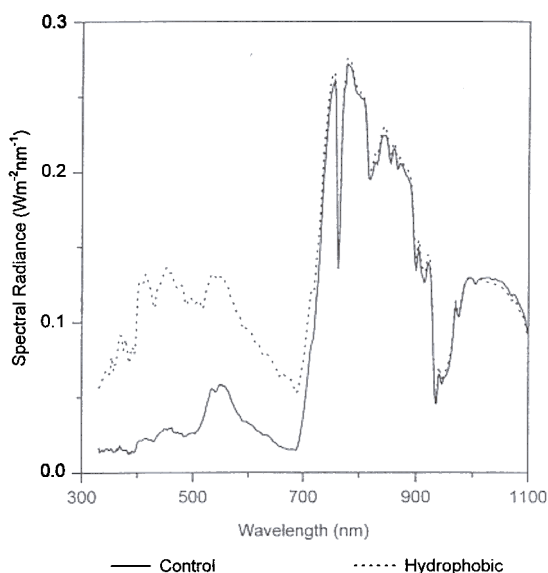


Fig. 3. Spectral radiance of solar radiation reflected from an untreated pear canopy and a canopy treated with hydrophobic particles.

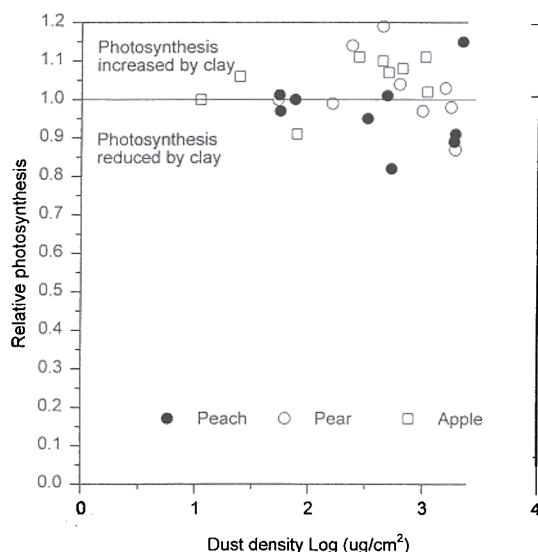


Fig. 4. Relative photosynthetic rates in apple, peach and pear after applying increasing levels of hydrophobic particles to their leaves.

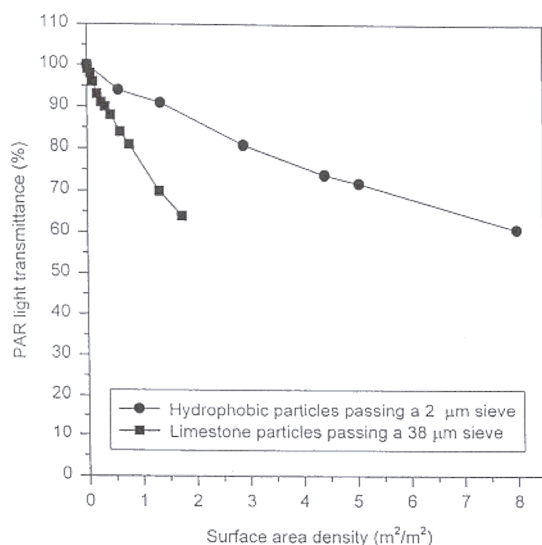


Fig. 5. Transmission of photosynthetically active radiation (PAR) through treated surfaces.

divided particle size (Fig. 5). Natural dusts typically range in diameter from 5 to 100  $\mu\text{m}$  (Farmer 1993). In contrast, the hydrophobic particle of M96 used in this study are  $\leq 2 \mu\text{m}$  in diameter. The neutral effect of the hydrophobic particle film on leaf photosynthesis was evident in a field study where mature peach trees were treated weekly with M96 and no reduction in yield or shoot growth occurred (Table 9). Canopy temperature was also significantly reduced (Table 9) probably by increased reflection of infrared radiation at the leaf surface (Fig. 3). Baradas et al. (1976) found that dusting soybean with a white kaolin mineral decreased transpiration and water use because both global and long-wave radiation reflection were increased and the emissivity of the leaves had decreased. Similarly, Stanhill et al. (1976) demonstrated reduced water use in grain sorghum as a result of treatment with kaolin whitewashes and his results were duplicated by Moreshet et al. (1979) on dryland cotton. Basnizki and Evenari (1975) correlated increased stomatal diffusive resistance as a result of whitewash coverings with reduced water use. Reducing net radiation at the fruit surface is also important in reducing sunburn and heat stress effects. Lipton and Matoba (1971) and Lipton (1972) reduced internal fruit tissue temperatures of melons with whitewash applications and improved fruit quality. Based on our studies, hydrophobic par-

ticle film applications have had neutral effects on plant yield and vigor, but may improve plant physiological processes by reducing heat and water stress.

In conclusion, we found that a hydrophobic particle film based on the inert mineral kaolin can suppress disease and insect damage in fruit crops. Combining the attributes of hydrophobicity and finely divided particles together into a particle film material invokes a number of mechanisms for suppression of both arthropod pests and diseases of plants. Diseases can be prevented by enveloping the plant with a hydrophobic particle film barrier that prevents disease inoculum or water from directly contacting the leaf surface. Thus, many fungal and bacterial pathogens that require a liquid film of water for disease propagule germination are prevented. Arthropods can be repelled from, or infestations suppressed on, a plant coated with a hydrophobic particle film barrier by making the plant visually or tactually unrecognizable as a host. Furthermore, insect movement, feeding, and other physical activities can be severely impaired by the attachment of particles to the arthropod's body as they crawl upon the film. We elucidated the main mechanisms associated with arthropod pests and diseases of plants (Fig. 6). There are likely other mechanisms involved that will be discovered as hydrophobic particle films are studied further on other plants and their associated pests. Other key features of the hydrophobic particle film are that it reduces heat stress by reflecting sunlight with its bright white color, and does not affect plant photosynthesis or productivity because of the porous nature of the film.

We introduced a unique concept, hydrophobic particle film technology, that represents the combined synthesis of knowledge on hydrophobic films, physical particle barriers, and white reflective plant surfaces to suppress arthropod pests and diseases. Our results suggest that hydrophobic particle films could offer broad spectrum protection against arthropod pests and diseases in certain agricultural crops. Application of dusts is not a common commercial practice, although new technologies are being developed to efficiently apply dry materials and prevent drift (Wilson et al. 1995). However, we have made rapid progress on developing a formulation that enables hydrophobic particles to be mixed into water and sprayed using conventional commercial spray equipment.

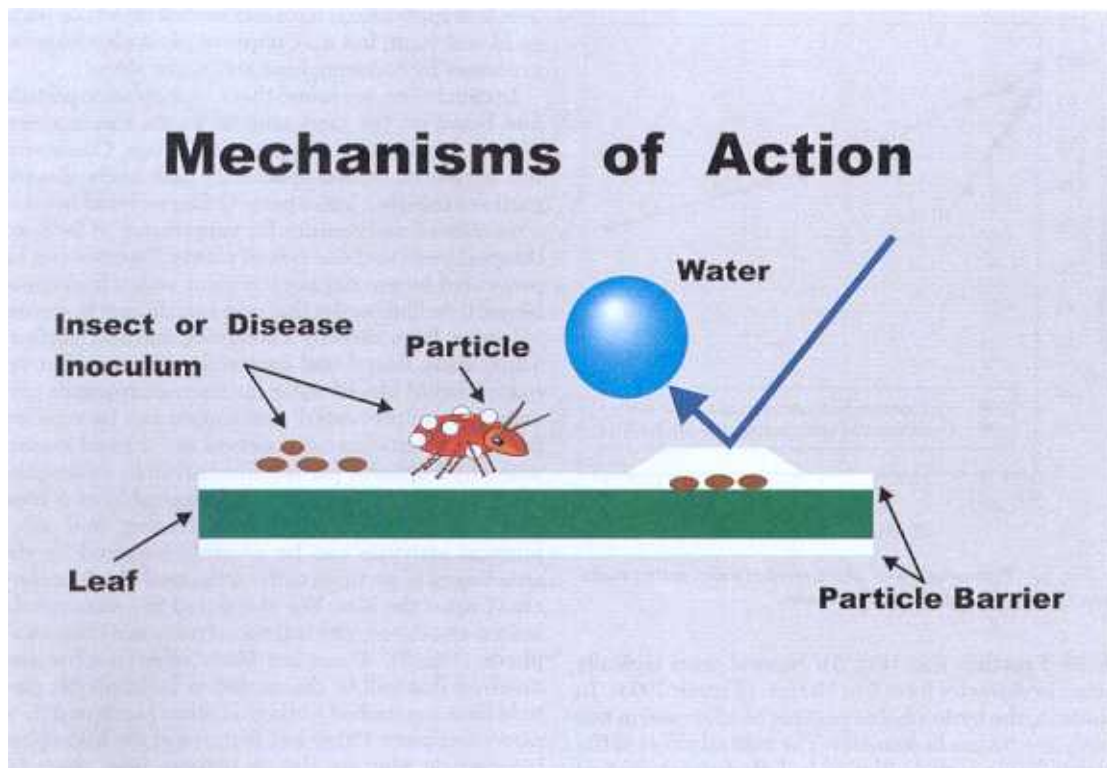
The use of hydrophobic particle films in crop protection will require the adoption of new pest control strategies aimed at pest prevention. The hydrophobic particle film acts primarily as a protective barrier and

Table 9. Canopy temperature, fruit yield and shoot length for 8-yr-old Loring peach after weekly dust applications of M-96-018 kaolin hydrophobic particles during the season, Kearneysville, WV, 1995

Treatment	Temp. differential $^{\circ}\text{C}^a$	Yield (kg)/tree		Shoot length of annual growth	
		Harvester	Loring	Harvester	Loring
Hydrophobic particle film	$-3.1 \pm 0.4a$	$14.4 \pm 1.1a$	$17.1 \pm 1.8a$		
Conventionally sprayed	$-2.4 \pm 0.4b$	$15.2 \pm 1.5a$	$16.6 \pm 1.9a$		

Means within a column followed by the same letter are not significantly different (LSD,  $P > 0.05$ ).

<sup>a</sup> Tree canopy temperature minus air temperature averaged over 3 trees per replicate, 4 replications for 5 dates of sampling.



**Fig. 6.** Mechanisms of arthropod pest and disease suppression in plants. Arthropods can be repelled from or infestations suppressed on a plant coated with a hydrophobic particle film barrier by making the plant visually or tactually unrecognizable as a host. Insect movement, feeding, and other physical activities can be severely impaired by the attachment of particles to the arthropods body as they crawl upon the film. Diseases can be prevented by enveloping the plant within a hydrophobic particle film barrier that prevents disease inoculum from directly contacting the leaf surface. In addition, the particle barrier inhibits disease by preventing a liquid film of water from directly contacting the leaf surface. Thus, many fungal and bacterial pathogens that require a liquid film of water for disease propagule germination are prevented.

must be in place before pest infestations result rather than when a critical economic threshold of a pest infestation is reached. Both the kaolin particle and the proprietary waterproofing surface treatment are generally regarded as safe under the U.S. Food and Drug Administration Food Chemical Codex and are considered an indirect food additive under 21CFR186.1256. Kaolin is classified only as a nuisance dust by the American Conference of Governmental Industrial Hygienists. Therefore, this concept offers an alternative pest management strategy with improved safety to pesticide handlers and the overall environment.

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